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(21) International Application Number: PCT/CA98/00374 (22) International Filing Date: 21 April 1998 (21.04.98) (30) Priority Data: 60/045,104 25 April 1997 (25.04.97) US (71) Applicant (for all designated States except US): ENVIROLUTIONS INC. [CA/CA]; 26 Burford Road, Hamilton, Ontario L8E 3C7 (CA). (72) Inventors; and (75) Inventors/Applicants (for US only): WILLIAMS, Kenneth, Roger [US/US]; P.O. Box 159, Landenberg, PA 19350 (US). CONWAY, Marlene, Elizabeth [CA/CA]; 421 Elizabeth Street, Grimsby, Ontario L5M 3K9 (CA). (74) Agent: GALLOWAY, Warren, J.; Sim & McBurney, 6th floor, 330 University Avenue, Toronto, Ontario M5G 1R7 (CA).		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: POLYACRYLATE GEL FOR HORTICULTURAL USE (57) Abstract A polyacrylate for horticultural use. The polyacrylate is a polyacrylate of a divalent cation and has an absorption capacity index, as defined, in the range of 30-100. A horticultural composition comprising a polyacrylate that has been admixed with at least 200 times its weight of water. A method of forming a horticultural composition comprising admixing a polyacrylate with at least 250 times its weight of water, especially at least 350 times its weight in water. The polyacrylate is useful in, for example, the germination of seedlings, with plants or for transportation of plants or seedlings.		

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POLYACRYLATE GEL FOR HORTICULTURAL USE

The present invention relates to polyacrylate gel for horticultural uses, and its use therein, and in particular to modification of sodium polyacrylate superabsorbent polymer gels for horticultural use.

As used herein, absorption capacity index (ACI) is defined as: (wt of water saturated gel polymer - polymer dry wt)/polymer dry wt. The measurement of ACI is described below.

Plants and seedlings are normally germinated and grown in commercial operations dedicated to that business. However, the plants and seedlings are normally not offered to sale, or only a small percentage is offered for sale, at the location at which the plants and seedlings are germinated. It is common practice for plants and seedlings to be shipped to other locations involved in the retail of plants and seedlings to the public. In cities, a wide variety of stores will offer plants and seedlings for sale, including grocery stores, department stores, multi-purpose stores, hardware stores and many others. In addition, there are retail stores that specialize in the sale of horticultural products to the public. Alternatively, the plants and seedlings might be mailed or shipped directly to a person or entity e.g. a market gardener, that has ordered the plants or seedlings from the grower.

Adequate moisture supply is critical to roots during plant shipment and to plants whose growing medium is subject to long periods of moisture deficiency. Various superabsorbent polymer (SAP) gels have been offered commercially to address the problems of inadequate

moisture supply to roots. So called agricultural SAP chemicals, which are acrylamides or acrylamide copolymers, are non-ionic or have a very low anionic character. As a consequence of the non-ionic state, there tends to be a relative insensitivity to the presence of cations in the soil and hence the degree of swelling of the agricultural SAP tends to remain constant over repeated wet/dry cycles. In contrast, anionic SAP's which are sodium polyacrylate, tend to lose their ability to absorb large quantities of water in a cyclic wet/dry environment because of exchange of cations from the surrounding soil, particularly from clay soils.

During dry periods, sodium polyacrylate tends to condense and form crosslinks that inhibit re-swelling when it is re-wetted. Even when used in situations where a limited number of wet/dry cycles are experienced, sodium polyacrylate inhibits plant growth or in some cases is toxic to plants. This inhibition of plant growth or toxicity is believed to arise because the sodium ions in the sodium polyacrylate network are exchangeable and these ions are adsorbed by clay particles or tend to undergo exchange with cations on the surface of plant roots. The consequence is a condition that is analogous to an alkali soil, which generally tends to adversely affect or inhibit plant growth.

Sodium polyacrylate is the dominant superabsorbent polymer product in use today. Its major application is as a body fluid absorbent in hygienic disposal products, particularly in diapers. In such uses, rapid absorption of water is an essential requirement, and the polymer must have a strong affinity for water. It is estimated that over 90% of all superabsorbent polymer produced is

sodium polyacrylate. Such an economy of scale in its production means that the cost of sodium polyacrylate tends to be lower than that of superabsorbent polymers otherwise used for agricultural end uses. In addition
5 commercial processes are now available for recovery of superabsorbent polymer from hygienic disposal products, including diapers. Such a process is disclosed by M.E. Conway et al in U.S. 5,558,745.

A number of water absorbing substances have been
10 disclosed as additives for soil to provide a moisture source for plants. An important criteria for successful use is that the water absorbing substance must be capable of maintaining its water retention characteristics over a series of wet/dry cycles. Polyacrylamides appear to have
15 superior cyclic moisture retention characteristics than polyacrylates and commercial agricultural SAP's are polymers or co-polymers of acrylamide. U.S. 5,405,425 discloses that a sulphonyl-containing polyacrylamide shows superior retention of its ability to reabsorb water
20 during cyclic wet/dry treatment cycles.

Sodium polyacrylate gel offers potential for use in horticultural end uses, provided that the tendency of sodium polyacrylate to inhibit plant growth or be toxic to plants can be reduced.

25 Accordingly, the present invention provides a polyacrylate for horticultural use, said polyacrylate being a polyacrylate of a divalent cation and having an ACI in the range of 30-100, especially 40-100.

In a preferred embodiment, the polyacrylate is
30 obtained from a sodium polyacrylate by exchange of said sodium cation with said divalent cation.

In a further embodiment, the polyacrylate of the divalent cation has a water content of less than 50%.

In yet another embodiment, the divalent cation is an alkaline earth metal, especially calcium.

5 The present invention also provides a method for the preparation of a polyacrylate for horticultural use, comprising the steps of treating an alkali metal polyacrylate in aqueous solution with a water-soluble compound of a divalent cation, separating the gel so
10 formed from the aqueous solution and drying the gel to a moisture content of less than 50%.

 In preferred embodiments of the method, the water-soluble compound is a compound of an alkaline earth metal, zinc or iron(ferric), especially a calcium
15 compound e.g. calcium hydroxide or lime, calcium sulfate or gypsum, or calcium nitrate.

 In another embodiment, a nonionic water-soluble compound is added to the aqueous solution prior to filtering the gel e.g. urea or sucrose.

20 The present invention also provides horticultural use of the polyacrylate disclosed herein for the germination of seedlings, or for the transportation of plants or seedlings, the polyacrylate being used as a water-retentive medium.

25 The present invention is a polyacrylate gel for horticultural end-uses, although it could be more generally used in agricultural end-uses, depending on cost. It will be referred to herein as a soil additive.

 The soil additive is a polyacrylate and would normally
30 be formed from a sodium or potassium polyacrylate superabsorbent polymer (SAP) that has been treated in its gel state.

The superabsorbent polymer may be virgin polymer, but it is particularly intended that the superabsorbent polymer would be such polymer that has been recovered from another process. One example of such recovery is from used disposable diapers or other absorbent sanitary paper products, also referred to as personal care products, during processes for recycling and recovery of components of such processes for future use. One such process is that of the aforementioned U.S. 5 558 745.

10 The polyacrylate may be in a variety of forms before treatment as described herein.

Superabsorbent polymers that are acrylate polymers are normally cross-linked during the manufacturing process. Any cross-linking referred to herein is in addition to cross-linking that may have occurred in the processes for the manufacture of the polymer.

15

As a result of inherent water absorbent properties, superabsorbent polymers tend to swell on contact with water. In embodiments of the present invention, the super absorbent polymer, after treatment for use in the soil additive of the present invention, has an absorption capacity index (ACI) that is at least 30, e.g. in the range of 30-100, especially in the range of 50-80.

20

The ACI of an anionic superabsorbent polymer, such as the polyacrylate polymers, may be decreased by cross-linking of the polymer with cations. Examples of chemical compounds that may be added to an aqueous solution of the anionic superabsorbent polymer to effect cross-linking of the superabsorbent polymer include soluble salts of at least one of an alkali metal, an alkaline earth metal, aluminium, copper (II), iron (III) and zinc. However, the salts that may be added to effect

25

30

cross-linking of superabsorbent polymer for the present invention, including sodium polyacrylate and potassium acrylate, are salts of divalent cations, especially alkaline earth metal cations. Examples of such divalent
5 cations are magnesium, calcium, barium and zinc. Calcium is preferred.

Examples of the salts include calcium chloride, calcium nitrate, dicalcium phosphate, tricalcium phosphate, magnesium chloride, magnesium nitrate,
10 magnesium sulphate, barium chloride, barium nitrate, zinc sulphate and zinc nitrate. Calcium hydroxide may also be included to aid in deswelling the SAP.

The salts may be soluble in water or only partially so. In the latter case, ions in solution will be
15 absorbed, and the ions removed from solutions will be replenished from undissolved salt, according to solubility equilibria of the salt in the solution.

The amounts of cross-linking agent are preferably adjusted so that the absorption capacity index (ACI) of
20 the super absorbent gel polymer is at least 30, especially 30-100, as indicated above.

The particulate gel super absorbent polymer that has been treated as described herein is separated from the aqueous solution and subjected to drying procedures,
25 preferably in a heated air stream at about 60°C. In embodiments, drying is allowed to proceed until a solid with a moisture content of less than 50% is obtained, especially a moisture content of about 1-10% moisture. The solid is then ground to size for adding to soil.

30 A non-ionic water-soluble additive may be added to the gel before drying. The amount of the non-ionic water-soluble additive may be varied widely, but in

embodiments is up to 50% by weight of the dried solid, especially 10-40% by weight. Examples of such non-ionic water-soluble additives are urea and sucrose.

The non-ionic water-soluble additive, if used, may
5 be added in a separate step. However, it is preferred that the treatment and formation of the particulate form of the super absorbent polymer and addition of the non-ionic water-soluble additive be carried in one step. Growth promotion may also be achieved e.g. by addition of
10 such an additive or by utilizing a cross-linking agent that is in itself also a growth-promoting agent e.g. a nitrate.

The soil additive of the invention is added to soil, for instance by using techniques typically used for the
15 addition of fertilizers to soil. The amount of soil additive added to soil may be varied over a wide range of concentrations. The soil additive may be used as the only medium for the plant or seedling i.e. in the horticultural use e.g. the plant or seedling could be
20 grown in the soil additive per se, or the soil additive may be admixed with soil or an inert material. In embodiments of the present invention, the soil additive is used in admixture with soil, especially with soil as the major component for germination of seedlings and
25 used in higher concentrations, including used alone, with plants e.g. for the shipping of plants.

In other embodiments, the present invention provides a horticultural composition formed by admixing sodium polyacrylate with at least 250 times its weight of water,
30 preferably with an amount of water that is in excess of the amount of water that is absorbable by the sodium polyacrylate. A preferred amount is at least 350 times

its weight in water. Excess water is then removed e.g. by filtering or pressing. The sodium polyacrylate may then be used in horticultural end-uses e.g. for shipping of plants. Alternatively, the sodium polyacrylate may be
5 partially dried. For instance, the sodium polyacrylate may be dried so that at least 50% of the water absorbed into the solution polyacrylate remains in the sodium polyacrylate. Alternatively, at least 50% of the water is removed, especially so that the moisture content of
10 the sodium polyacrylate is less than 50% by weight. It is preferred that the water used in the treatment of the sodium polyacrylate contain divalent cations e.g. calcium or magnesium. Some municipal tap water is believed to contain sufficient of such ions, naturally-occurring in
15 the water, to be acceptable for such treatment.

Municipal water, or water from other sources, may contain additional naturally-occurring ions e.g. ferric ions.

For some uses, it is believed that addition of water and partial drying of the sodium polyacrylate will
20 provide an acceptable horticultural product e.g. for shipment of plants.

The invention discloses a way to modify a polyacrylate, especially a sodium or potassium polyacrylate, so that it may be used for, in particular,
25 horticultural end-uses, especially for plants and seedlings without the adverse effects of sodium or other alkali metal. As the soil dries out, the zone surrounding the super absorbent particulate better retains its moisture. Thus, it is believed that a more
30 desirable environment is created for the plant roots in these zones. Such environment is beneficial until such time as the plant or seedling is planted, providing a

more consistent supply of moisture, especially during long periods of moisture deficiency.

The absorption capacity index (ACI) test used herein was as follows: 1.0g of the dried particulate product was placed in 200 ml of water for a period of time. The resultant gel was collected on a fine mesh screen and the weight of the gel was measured, from which the ACI value was calculated. The procedure was repeated, after discarding the water not absorbed in the gel, using a further 200 ml of water and the ACI value was recalculated. This procedure was repeated for 5 or more cycles. This testing cycle was used as a simulation of the moisture behaviour found in soil. For instance, under wet soil conditions, where there is runoff and/or loss to the water table in the soil, the SAP should experience swelling similar to immersion in water. As the soil dries out, water diffuses out of the SAP along with trapped salts and it will reach the moisture content measured in the "gel" state. Nonetheless, it is understood that in actual conditions in a soil, further soil drying will also reduce the water content of the SAP, but this loss will be influenced by the osmotic forces developed in the soil. This was deemed to be outside the scope of measurement in laboratory tests used to assess the present invention.

The present invention is illustrated by the following examples.

EXAMPLE I

Germination of Blue Lake bush green beans was studied in three selected commercially-available superabsorbent polymer gels, as well as in an

experimental sodium polyacrylate gel containing a controlled release fertilizer.

It is known that beans have a low salt tolerance, (C. Bower and M. Fireman, "Saline and Alkaline Soils in Soil" 1957 Yearbook of Agriculture U.S. Dept. of Agriculture, Washington D.C. p. 288).

The gels were made up 1:100 with tap water.

TABLE I

Bean Seed Germination in 100% SAP Gel Media			
<u>SAP Type</u>	<u>Use</u>	<u>% Germination, 11 Days</u>	
Stockhausen, Fam	Hygienic Disposables	0	
Terasorb™ HB	Agricultural	83	
Alcosorb™	Agricultural	83	
60811 A	Sodium Polyacrylate Polymer	0	

The gels made from agricultural superabsorbent polymers, which are based on acrylamide polymers, yielded a high level of seed germination. In contrast, the superabsorbent polymer for hygienic disposables and the experimental gel, both of which are based on sodium polyacrylate systems, showed zero germination.

EXAMPLE II

The efficacy of chemically modified sodium polyacrylate SAP gels as a plant transplant medium were studied using Blue Lake bush bean seedlings grown for 17 days in Peter's Professional Potting Soil, Scots-Sierra Horticultural Products Co., Marysville, OH. The bare root seedlings were transplanted after two permanent leaves were developed on each specimen, into 6 oz clear polyester cups containing various SAP gels. The dry SAP

test items were mixed 1:400 with tap water and they were then filtered using a screen to remove unabsorbed water. After 15 days the seedlings were rated for new leaf growth after transplanting.

5 The gels used were as follows:

A - Alcosorb agriculture SAP

S - Stockhausen Fam - hygienic disposable SAP

X - Experimental SAP made by:

- 10 1. Mixing 10 g Stockhausen fam sodium polyacrylate in 4 liters of water, and filtering;
2. Mixing the filtrate from Step 1 with 2.35 g $\text{Ca}(\text{OH})_2$, and filtering;
- 15 3. Drying the filtrate from Step 2 to constant weight.

The results are reported in Table II.

TABLE II

20 $X_1 > X_2 > X_3 > S_1 = S_2 = S_3 > A_1 = A_2 = A_3 = X_4 > S_4 > S_5 = X_5 > A_4 > S_6 > A_5$

Of the five samples of the experimental gel in the test, the results for three were the best of all samples tested.

25 The data indicate that bean seedling leaf growth was best in the experimental gel, followed by leaf growth in the Stockhausen gel, with the smallest leaf growth being in the Alcosorb gel.

30 Good seedling leaf growth in the Stockhausen gel viz. a sodium polyacrylate, is believed to be a result of treatment with excess tap water. It is to be noted that in Example I, the seeds were planted directly in the

gels. In contrast, in Example II the gels were first swollen in an excess of water which was removed during a filtration step. The filtration step may have resulted in removal of sufficient sodium ions to render this product acceptable as a plant transplant medium.

EXAMPLE III

The effect of reduced degree of hydration of sodium polyacrylate gel on growth of bush bean seedlings was tested. In Example II, a ratio of sodium polyacrylate:water of 1:400 was used. In this example, the ratio was reduced in separate tests, to 1:200, 1:100 and 1:50.

Treatment of the bean seedlings followed the procedure outlined in Example II. Separate tests were conducted in which tap water and distilled water were used to hydrate the sodium polyacrylate.

TABLE III

Growth of Bean Seedlings Immersed in Sodium Polyacrylate Gels of Low Degrees of Hydration

NaPA:Water Ratio	Tap Water		Distilled Water	
	Seedling Appearance	Time (hrs)	Seedling Appearance	Time (hrs)
1:200	Slight wilting	224	Slight wilting	114
1:100	Moderate to total wilting	224	Moderate wilting	114
1:50	Leaves wilted or dead	224	Completely wilted	114

The results with sodium polyacrylate gels of lower degree of hydration are in sharp contrast to the results obtained in Example II in which all seedlings survived and continued to grow when placed in clay pots containing a soil mix. In this example, as the degree of gel hydration was reduced, seedling growth was increasingly inhibited.

Wilting was more rapid when distilled water was used for hydration than when tap water was used. This observation could be because the ACI of sodium polyacrylate gels is believed to be reduced in the presence of cations present in the hydrating media i.e. tap water contains more cations than distilled water. The wilting characteristic might be primarily caused by a loss of water from the plant roots to the incompletely hydrated gel or from a substitution of cations in the roots with sodium ions from the gel, or for other reasons.

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EXAMPLE IV

The addition of a divalent compound to a sodium polyacrylate gel must be sufficient to replace most or all of the sodium ion attached to the polymer chain. However, use of excess divalent cations will prevent the resulting product, after it has been dried, from reabsorbing significant quantities of water. Such a product would be expected to have little utility as a plant transplant or preservation medium.

The reswelling characteristics of a processed recycled sodium polyacrylate gel can be determined from

the ACI value after the divalent deswelling compound is added to the swollen gel.

A gel was made by adding 6.0 g agricultural gypsum [82.2% $\text{CaSO}_4 \cdot \text{H}_2\text{O}$] to 10 g of Stockhausen Fam SAP, followed by 5.0 g of urea. The ACI was then determined, and found to be 29.6. After drying, this material which then had an ACI of 23, did not reswell adequately in tap water.

In contrast, the experimental gel described in Example II had an ACI value of 61 after addition of Calcium hydroxide. After drying and reswelling in tap water, this gel had an ACI of 91.

EXAMPLE V

A gel was formed from 3.0 g of sodium polyacrylate mixed in 405 g of tap water. To this gel was added a solution of 0.9 g of ferric chloride hexahydrate in 45 g of water. The gel dewatered to yield an ACI value of 82. This result indicates that a large quantity of sodium ion present in the original super absorbent polymer had been replaced by ferric ion. When this product was dried and water was reintroduced [1.0 g dry solid/200ml water], an ACI value of 30 was obtained. Rewetting the filtered gel with 200 ml quantities of water yielded succeeding ACI values of 43 and 51. Thus the swelling characteristics are similar to those found when calcium compounds are mixed with SAP.

EXAMPLE VI

The swelling characteristics observed in the previous examples using sodium polyacrylate polymer were also observed with potassium polyacrylate polymer. In this example, 4.0 g of Aridall™ 1460 (a potassium

polyacrylate super absorbent polymer, produced by the Chemdal Corp.) was added to a ferric chloride solution composed of 0.4 g of ferric chloride hexahydrate in 240 g of tap water. The Aridall 1460 polymer absorbed all of the ferric chloride solution. Next 0.8 g of ferric chloride hexahydrate dissolved in 50 g of water was added to the gel and rapid dewatering occurred. The filtered gel had an ACI of 41. To this was added 10.0 g of urea pellets which dissolved into the gel structure. After drying, the resulting product had similar swelling characteristics when added to water (1.0 g dry solids/200 ml tap water) observed in Examples IV and V. The consecutive ACI values were 14, 23 and 27.

CLAIMS:

1. A polyacrylate for horticultural use, said polyacrylate being a polyacrylate of a divalent cation or iron III and having an absorption capacity index, as defined herein, in the range of 30-100.

2. The polyacrylate of Claim 1 in which the polyacrylate is a polyacrylate of a divalent cation.

3. The polyacrylate of Claim 2, in which the polyacrylate is obtained from a sodium polyacrylate by exchange of said sodium cation with a divalent cation.

4. The polyacrylate of Claim 2 in which the polyacrylate with the divalent cation has a water content of less than 50% by weight.

5. The polyacrylate of Claim 2 or Claim 3 in which the divalent cation is an alkaline earth metal.

6. The polyacrylate of Claim 5 in which the divalent cation is calcium.

7. The polyacrylate of any one of Claims 1-6 having an absorption capacity index in the range of 40-100.

8. A horticultural composition comprising a polyacrylate that has been admixed with at least 200 times its weight of water.

9. The composition of Claim 8 in which the polyacrylate has been admixed with water in excess of the amount that may be absorbed by the polyacrylate, said excess water subsequently being removed.

10. The composition of Claim 9 in which the water content is less than 50% by weight of the amount of water absorbable by the polyacrylate.

11. The composition of Claim 10 in which the polyacrylate contains less than 50% by weight of water.

12. The composition of Claim 9 in which the water is water containing naturally-occurring divalent cations.

13. The composition of Claim 9 in which divalent cations have been added to the water.

14. The composition of any one of Claims 8-13 in which the polyacrylate is sodium polyacrylate.

15. A method for the preparation of a polyacrylate for horticultural use, comprising the steps of treating an alkali metal polyacrylate in aqueous solution with a water-soluble compound of a divalent cation or iron III, separating the gel so formed from the aqueous solution and drying the gel to a moisture content of less than 50%.

16. The method of Claim 15 in which the dried gel so obtained has an absorption capacity index, as defined herein, in the range of 30-100.

17. The method of Claim 16 in which the absorption capacity index is in the range of 40-100.

18. The method of Claim 16 in which the water-soluble compound is a compound of an alkaline earth metal or zinc.

19. The method of Claim 18 in which the compound is a calcium compound.

20. The method of Claim 19 in which the compound is calcium hydroxide or lime.

21. The method of Claim 19 in which the compound is calcium sulfate or gypsum.

22. The method of Claim 19 in which the compound is calcium nitrate.

23. The method of Claim 15 in which a nonionic water-soluble compound is added to the aqueous solution prior to filtering the gel.

24. The method of Claim 23 in which the nonionic water-soluble compound is urea or sucrose.

25. A method of forming a horticultural composition comprising admixing a polyacrylate with at least 250 times its weight of water.

26. The method of Claim 25 in which the polyacrylate is admixed with at least 350 times its weight in water.

27. The method of Claim 25 in which the polyacrylate has been admixed with water in excess of the amount that may be absorbed by the polyacrylate, said excess water subsequently being removed.

28. The method of Claim 27 in which the water content of the polyacrylate so obtained is at least 50% by weight of the amount of water absorbable by the polyacrylate.

29. The method of Claim 27 in which the polyacrylate so obtained contains less than 50% by weight of water.

30. The method of Claim 25 in which the water is water containing naturally-occurring divalent cations.

31. The method of Claim 25 in which the divalent cations have been added to the water.

32. The method of Claim 25 in which the polyacrylate is sodium polyacrylate.

33. Horticultural use of a polyacrylate of Claim 1 for the germination of seedlings, with plants or for transportation of plants or seedlings.

34. Horticultural use of a composition of Claim 8 with plants, or transportation of plants or seedlings.

35. A horticultural composition comprising 10-100% by weight of sodium polyacrylate and 0-90% by weight of horticulturally-acceptable material, said sodium polyacrylate containing water and having been treated so that the resultant horticultural composition sustains growth.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/CA 98/00374

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C09K17/22 C09K17/48 A01G31/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C09K A01G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 3 580 879 A (E.HIGASHIMURA) 25 May 1971 see column 1, line 15 - line 34 see column 3, line 71 - column 4, line 11 see column 3, line 19 - line 34 ---	1,2,5,6
Y	EP 0 521 355 A (MITSUBISHI PETROLEUM COMP.) 7 January 1993 see page 2, line 50 - page 3, line 9 see page 3, line 34 - page 4, line 39 ---	1
Y	US 5 065 822 A (E.E.MILLER) 19 November 1991 see column 2, line 22 - column 3, line 54 see claims 1,3-5 --- -/--	1

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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